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Decomposition of Halogenated Organic Materials by Electrified Microheterogeneous Catalysis

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DECOMPOSITION OF HALOGENATED ORGANIC MATERIALS BY ELECTRIFIED MICROHETEROGENEOUS CATALYSIS

INTRODUCTION

Polychlorinated biphenyl (PCB) species were in wide commercial use from 1929 to the late 1970's due to their low flammability, high dielectric constant, and unusually high chemical and thermal stabilities. PCB's sold under the tradename Aroclor are mixtures typically with five to seven chlorines per biphenyl moiety, and there are 209 different PCB compounds and isomer combinations theoretically possible [1]. The chemical and physical properties that made them attractive commercially are the reason for the difficulty in their destruction; disposal is further complicated by the number of PCB compounds and isomers in each Aroclor sample.

Chlorinated aromatics are presently destroyed through incineration at temperatures in excess of 1000 °C. This is a highly efficient method for PCB disposal (>99% destruction efficiency), but there exists the potential for trace contaminant emission and/or dioxin formation due to incomplete combustion [2]. Alternative destruction methods are under active investigation and include electrocatalysis [3-6], photocatalysis [7,8], bioremediation [9], and use of chemical reagents [10] and supercritical water [11]. Problems for these methodologies range from the technical to the practical; for example, the need to add undesirable chemical reagents [3-6,10] to low economic efficiencies [7,8].

We have begun experiments to demonstrate that chemical reactions typically driven with temperature can be controlled to the same extent with electrical energy as the driving force [12]. This paper describes our preliminary experiments designed to evaluate the efficacy of dispersion electrolysis as an alternative to incineration for destruction of halogenated materials. Through use of a voltage field of $<100 \text{ V cm}^{-1}$ and a NaY zeolitic catalyst suspension, we have found that chlorobenzene, Aroclor 6050, and CFC-113 can be destroyed in deionized water at 0 °C [12c].

RESULTS AND DISCUSSION

Chlorobenzene. We have performed a series of survey reactions to test the generality of our process for chlorobenzene degradation with a variety of catalysts, both zeolitic and non-zeolitic. Shown in Table 1 is a qualitative summary of these experiments. Our initial interest was to test for dechlorination and this was performed qualitatively with AgNO_3 ; we also made visual observations of both the aqueous and non-aqueous phases. As our analytical capabilities increased, the aqueous phases were assessed by ion chromatography and the non-aqueous phases were further analyzed by GC-MS to determine the number of products formed and to elucidate their identities by their fragmentation patterns. *It is important to note that these are preliminary findings only and no one experiment should be given too much weight; as a group of experiments, however, the trends are discernable.*

With the exception of Dowex-50X and NaOH, all of the catalysts employed were white powders, and upon suspension in deionized water formed a milky-white color. In general, we have found that a successful reaction is one where the cell contents (i) change color to yellow-brown, (ii) become increasingly frothy (presumably due to the formation of reaction products with surfactant properties), (iii) test positively for chloride, and (iv) upon extraction in diethyl ether result in a colorless non-aqueous phase which can be evaporated to dryness (the boiling point for diethyl ether is 35°C and that for chlorobenzene is 132°C). Upon inspection of Table 1, it can be seen that this is the case with only LY, NaY, and KY. While NaX also fulfilled these criteria, the destruction was not judged as complete; upon inspection of the cell contents at the conclusion of the reaction, two phases were clearly present. Although the non-aqueous phase was not analyzed by GC-MS, a strong and characteristic benzene odor was noticed; this suggests that an alternate reaction path was taken. Catalyst CaY had little effect on chlorobenzene, whereas HY, NaA, KL, and mordenite zeolites directed the reaction in yet another mechanistic route as observed by the yellow non-aqueous phases. These results suggest that it is the zeolite, and not the exchangeable cation, that is more important in influencing the chlorobenzene degradation mechanism.

The controls and non-zeolitic catalysts are listed in the last six entries of Table 1. Use of NaY without a applied voltage field yields no reaction. Zeolites are capable of ion exchange [13], and when NaY is placed in deionized water the sodium ions within the zeolite are exchanged for the protons in water which arises from the autoprotolysis of water, as described in Equations (1) and (2) (see Experimental section).

Table 1. Visual Observations of Chlorobenzene Reaction as a Function of Catalyst Identity.^a

<i>Catalyst</i>	<i>Aqueous Phase^b</i>	<i>Frothing?^b</i>	<i>Cl⁻ Present?^c</i>	<i>Ether Extract^d</i>
HY	Light Yellow	No	Minimal	Yellow Oil
LiY	Yellow-Brown	Yes	Yes	Colorless
NaY	Yellow-Brown	Yes	Yes	Colorless, 0 Products
KY	Yellow-Brown	Yes	Yes	Colorless
CaY	Colorless	No	No	Chlorobenzene
NaX	Yellow-Brown	Yes	Yes	Colorless
NaA	Yellow-Brown	Yes	Yes	Yellow Oil
KL	Yellow-Brown	Yes	Yes	Yellow Oil
Mordenite	Yellow-Brown	No	Yes	Yellow Oil, 14 Products
NaY, 0 V	Colorless	No	No	Chlorobenzene
0.2 mN NaOH	Colorless	No	No	Colorless, 4 Products
Dowex-50X	Light Yellow	No	Minimal	Chlorobenzene
γ -Al ₂ O ₃	Yellow-Brown	No	Yes	Yellow Oil, 7 Products
SiO ₂	Light Yellow	No	Yes	Yellow Oil, 7 Products
Kaolin	Light Yellow	No	Yes	Yellow Oil, 8 Products

Table 1 Notes

- a. All reactions were performed at 1-10 °C with an initial chlorobenzene concentration of 49 mM and at applied *dc* voltages of 20-30 V. The length of each reaction was 60-90 minutes.
- b. Observation after filtration of solid catalyst from aqueous phase.
- c. Based on formation of AgCl upon addition of AgNO₃; chloride was later confirmed by ion chromatography (IC). In addition, 2-6 additional unidentified anions were detected by IC.
- d. The reactions that were analyzed by GC-MS are denoted by entries for the number of products formed. These species have been tentatively identified and include chlorobenzoquinone, chloro-1,2-benzenediol, chloro-1,3-benzenediol, 1,2-benzenediol, 1,3-benzenediol, benzoquinone, ethenylbenzene, and 2-methyl-2-pentenal.

Our typical experimental conditions (a suspension density of 50 mg NaY in 20 mL H₂O) generate sodium concentrations of 100-600 μ M as measured by atomic absorption spectroscopy [14]. Direct electrolysis of chlorobenzene at the platinum feeder electrodes in dilute NaOH electrolyte is thus possible. As shown in Table 1, electrolysis in 0.2 mV NaOH solution has little effect on chlorobenzene, although four products were detected in trace quantities. Use of the sodium form of Dowex-50X, an organic cation-exchange resin, as the catalyst also resulted in minimal reaction. Lastly, the solids γ -Al₂O₃, SiO₂, and kaolin function as both dechlorination and oxygenation catalysts as observed by the positive chloride-ion results and by the formation of the yellow non-aqueous extract; subsequent GC-MS analysis of the ether phase shows both chlorinated and non-chlorinated benzene oxygenates as the major products.

Aroclor 6050. Based on the results from chlorobenzene degradation, we chose a NaY catalyst for decomposition of Aroclor 6050. However, unlike the experiments with chlorobenzene, it was necessary to employ a co-solvent (acetone) to form a proper dispersion with Aroclor 6050, a viscous yellow oil. If a co-solvent is not used, the Aroclor simply will not disperse but rather clumps together at the bottom of the cell - even with high dispersion flow rates of >200 cc min⁻¹. Conversely, we found that water is a necessary component for the degradation reaction as the process will not work in 100% acetone. Use of 3:1 acetone-to-water ratio did not completely dissolve the Aroclor sample, but satisfactorily suspended enough so that a proper dispersion could be maintained between the feeder electrodes.

After the NaY was ion-activated (see Experimental section) in the acetone-water mixture, 0.2 mL (200-400 mg) Aroclor 6050 was added to the dispersion cell at 0 V. The resulting solution resembled a "lava lamp" in that small clumps of the Aroclor rose to the top of the solution via the dispersion process, but then coalesced to form larger clumps, which fell to the cell bottom; this process was infinitely repeated. A voltage of 30 V was applied and after ~ 30 minutes the cell contents were observed to begin frothing and the solution color began to darken in color: yellowish initially, gradually becoming a dark yellow-green. It is noteworthy that the 30-minute induction period is not only repeatable for Aroclor 6050 degradation, but a similar 20-minute induction period was observed for reaction of chlorobenzene. Simultaneously with the frothing and observed color changes, the cell current begins a gradual increase. This increase continued for ca. two hours and then attained a steady-state value; the cell temperature did not increase and was maintained at <10 °C throughout the course of the reaction. The increase of the cell current to a larger steady-state value, which was also observed with chlorobenzene degradation, is presumably due to the cell contents becoming more ionically conductive with time - some of this increase can certainly be attributed to the creation of chloride ions via dechlorination.

Upon product workup, the aqueous phase visually tested positive for a greater amount of AgCl relative to that observed after chlorobenzene degradation. The non-aqueous ether extract was a dark

brownish-violet color and was analyzed by GC-MS, NMR, and IR. Shown in Figure 1 are the GC-MS chromatograms for Aroclor 6050 dissolved in diethyl ether before dispersion electrolysis at 30 V in a NaY suspension and for the ether extract of the dispersion after reaction. After reaction, the GC-MS shows the near-complete loss of many of the higher molecular weight congeners from the original sample, with subsequent formation of numerous lower molecular-weight species. Likewise in Figure 2 the 90-MHz ^1H -NMR results are shown for Aroclor 6050 dissolved in CDCl_3 before and after decomposition. The ^1H -NMR for the as-received Aroclor 6050 shows a single peak (with two spinning side bands) at ca. 7 ppm (relative to tetramethylsilane) that is indicative of a starting material with only aryl protons. After reaction, the aryl proton signal is broadened and attenuated, whereas several new upfield peaks are observed that could arise from alkyl and olefinic protons, possibly due to a ring-opening process. This is further substantiated by IR results, and these are exhibited in Figure 3. The spectrum for neat Aroclor 6050 (Figure 3: Before reaction) shows a sharp set of bands between 3000 and 3100 cm^{-1} due to stretching vibrations of the aryl C-H. After reaction, the IR exhibits a strong, broad peak at 3200-3500 cm^{-1} , due to carboxylic acid C-O and/or alcohol O-H stretching, and another strong, broad peak at 1700-1750 cm^{-1} , due to ester formation. A degradation mechanism which incorporated dechlorination, ring cleavage, and subsequent oxidation would be consistent with our GC-MS, NMR, and IR results.

As with the controls for chlorobenzene, if Aroclor 6050 is simply allowed to be stirred for 28 hours in the presence of NaY catalyst, there is no reaction. Similar results are obtained if the Aroclor-NaY suspension is refluxed at 57 °C for 29 hours: no reaction. When the platinum anode and cathode are replaced with 316-stainless steel, there is no significant reaction with chlorobenzene or Aroclor 6050; however, ion-activation of NaY at stainless steel is identical to that at platinum. It is not obvious at this point what the degradation mechanism is, but it is clear that for decomposition to proceed, both the catalyst and an applied voltage are necessary. Moreover, successful degradation is dependent on catalyst and electrode material selection.

CFC-113. Due to the low boiling point of this compound (47 °C) and the design of the dispersion cell, a larger amount of CFC-113 was added initially so that it did not entirely volatilize before any reaction could occur. NaY was again used and after its ion activation, 1.6 g (0.42 M) 1,1,2-trichlorotrifluoroethane was added to the dispersion cell; a minimal He flow rate of ca. 100 cc min^{-1} was employed to reduce volatilization as much as possible. A dry ice/acetone trap was employed to collect and measure the unreacted CFC-113. After application of 20 V for 35 minutes, over 50% of the CFC-113 had escaped the cell unreacted. However, the AgNO_3 test resulted in a precipitate, presumably AgF and AgCl ; fluoride was additionally identified by the qualitative glass-etch test [15]. Ion chromatography later confirmed the presence of both chloride and fluoride ions.

SUMMARY

In summary, the alkali-exchanged type-Y zeolites appear to direct the degradation of chlorobenzene and Aroclor 6050 more completely than the other zeolitic and non-zeolitic catalysts employed. In addition to C-Cl and C-F bond breakage, there is evidence for aromatic ring opening and subsequent oxygenation to alcoholic, carboxylic acid, and esteric species. Non-type-Y zeolites and non-zeolitic catalysts appear to function as both dechlorination and oxygenation catalysts in that a mix of halogenated and dehalogenated benzene oxygenates are formed during chlorobenzene decomposition. As our control experiments show, direct electrolysis is not an effective mechanism for decomposition or product formation in these low ionic strength electrolyte solutions. Finally, an applied *dc* voltage has been found to be necessary for decomposition to proceed; no reaction occurs when the halogenated organic material is placed in contact with the listed catalysts without the applied voltage, even at elevated temperatures.

It is important to point out that oxygen was not deliberately added to the dispersion cell in any of these experiments, yet oxygenated products from the degradation of chlorobenzene (in the presence of non-zeolitic catalysts) were observed to form. Voltages of the magnitude employed in this work, in dilute electrolyte, are sufficient to generate oxygen *in situ* at the anode during the reaction. Future experiments will assess the use of oxygen as a co-reactant on the reaction mechanism; alternatively, air may not only be sufficiently oxidizing in itself, but also more economically attractive.

EXPERIMENTAL

Catalysts. Sodium-exchanged type Y zeolite was obtained in the powder form (Strem) and used as received. Li- and K-exchanged type Y zeolites were prepared through four sequential ion exchanges [16] of NaY in 0.1 *F* LiCl or 0.1 *F* KNO₃ solution, respectively; CaY was analogously prepared from CaCl₂ solutions. The proton form of type Y (HY) was prepared by calcination of NH₄Y (Strem) in flowing oxygen at 400 °C. Type X (sodium form) was obtained from the Linde Division of Union Carbide and type L (potassium form), type A (sodium form), and mordenite (sodium form) synthetic zeolites were gifts from UOP. 1 wt% Pt⁰-NaY was prepared [17] following the general procedure of Gallezot [18]. All zeolite powders were stored in glass or plastic hydration chambers containing saturated NH₄Cl solution to ensure complete hydration prior to use. Non-zeolitic catalysts employed include kaolin powder (J. T. Baker), silica gel (Aldrich), Dowex-50X cation-exchange resin (Dow), γ -Al₂O₃ and 1 wt% Pt⁰-Al₂O₃ (Aesar), and NaOH (Fluka). All solid particles were $\leq 44 \mu\text{m}$ in size.

Reagents. Chlorobenzene and 1,1,2-trichlorotrifluoroethane (Aldrich) were both HPLC grade and used as received. Aroclor 6050 was obtained from the Chemistry Division at the Naval Research Laboratory. All water used for these experiments was obtained from a Millipore water purification system with an initial

resistivity $\approx 18 \text{ M}\Omega\text{-cm}$. Helium (Air Products) was employed as the dispersion gas; a minimum flow rate for the proper dispersion of zeolites has been found to be $80\text{-}100 \text{ cc min}^{-1}$.

Dispersion Cell. A schematic representation of the glass dispersion cell used for these experiments is shown in Figure 4. The outer portion of the cell is comprised of a female 34/45 glass joint and a 3-cm diameter fine-porosity glass frit connected through a pyrex cylinder for a total cell height of ca. 20 cm. The insert assembly is comprised of a male 34/45 glass joint connected to a pyrex cylinder sealed at the bottom. Note that when the cell is assembled, there is a 3-4 mm gap between the glass frit and the bottom of the insert assembly. Also shown is an exit port for gases that can serve as the location for on-line product analyses. Initial experiments were performed by immersion of the cell into an ice-water bath, but a water jacket now surrounds the outer reactor cylinder (not shown), to maintain constant temperature. Internal cell temperature is monitored with a thermometer in the well of the insert assembly. Platinum metal foils are employed as the anode and cathode and are spot welded into cylindrical forms and fitted into the cell as shown in Figure 4. The height of the electrodes is 5 cm and the gap between the two is 0.4 cm. When the cell is assembled, the volume of solution required to fill the cell to a level just above the electrodes is ca. 20 mL.

Instrumentation. A dc power supply (Sorensen Model DCR-600-1.5B or Kepco Model JQE) is used to apply a constant voltage across the cell. The cell current is determined by measurement of the voltage drop across a 10.2Ω , 160-W rated resistor placed in series with the dispersion cell (see Figure 4). Early product analyses were performed with a Finnegan ion-trap detector connected to a Hewlett-Packard 5890 gas chromatograph; products were separated on a 50 m x 0.2 mm Hewlett-Packard OV-101 column. Subsequent qualitative analyses were performed with a Finnegan TSQ-70 triple-quadrupole mass spectrometer connected to a Varian 3400 gas chromatograph; a 30 m x 0.32 mm J & W DB-5 column was used to separate products. Ion chromatography was performed with a Dionex modular system with conductivity detection; product anions were separated on a Dionex Ion Pack HPIC-AS4A column with bicarbonate buffer as eluent.

Procedure. A typical experiment is performed by first suspending 50 mg of catalyst in 20 mL deionized water. The suspension is then transferred to the electrochemical cell and the dispersion is maintained by a flow of inert gas ($>80 \text{ cc min}^{-1}$). After the cell contents have been thermally equilibrated to $0\text{-}5^\circ\text{C}$ the dispersion is "ion-activated," a term we use to describe the process of applying a dc voltage to the catalyst suspension while monitoring the current as a function of time [14]. This process appears to be a requirement for the zeolite-based catalysts, for which we obtain our best results, in that the subsequent chemical degradation reactions do not appear to be as facile at a non-ion-activated zeolite catalyst. When the ion-activation is complete (typically 10-20 minutes), the cell voltage is removed and the halogenated

organic material is added to the cell; the voltage is re-applied and cell current monitored as a function of time. Visual observations of the cell reaction and temperature are also recorded throughout the experiment. At the conclusion of the reaction, the cell contents are transferred to a separatory funnel and washed three times with diethyl ether. Upon separation of the two phases the catalyst is filtered from the aqueous phase, whereas the ether extract is dried over anhydrous MgSO_4 , filtered, and concentrated with the aid of a roto-evaporator.

Ion-Activation of Zeolite Catalysts. We define "ion activation" as the procedure by which a *dc* voltage is applied to a zeolite suspension prior to addition of any chemical reagents. As shown in Figure 5, a peak in the current-time curve is observed. The kinetics of this process are dependent on the identity of the zeolite, the charge-compensating cation in the zeolite, and on the magnitude of the applied *dc* voltage [14]; the latter is demonstrated in Figure 5 for applied *dc* voltages of 10, 20, and 30 V. Although the physical-chemical processes which occur are complex and not yet fully understood, we believe that the peak is related to (i) coupling of the water self-ionization and sodium-proton exchange reaction - as seen in Equations (1) and (2),



with a subsequent perturbation of the kinetics for these processes when the sodium-compensated aluminosilicate zeolites are in the presence of an applied voltage field [13], and (ii) an increased conductance of the zeolites' external surface [19].

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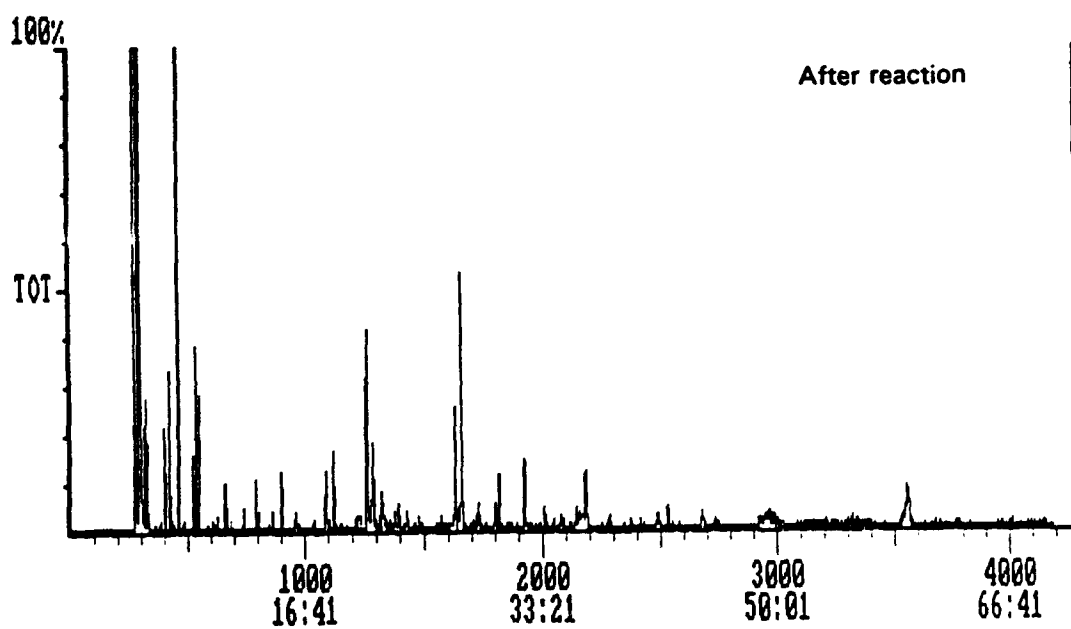
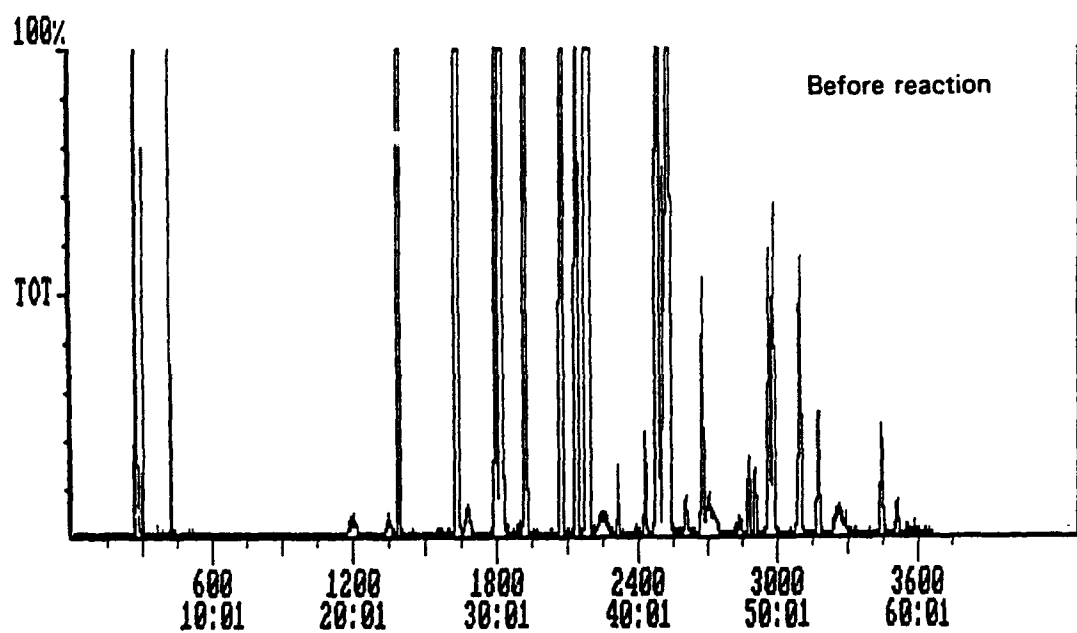


Figure 1. GC-MS chromatograms of Aroclor 6050 in diethyl ether.

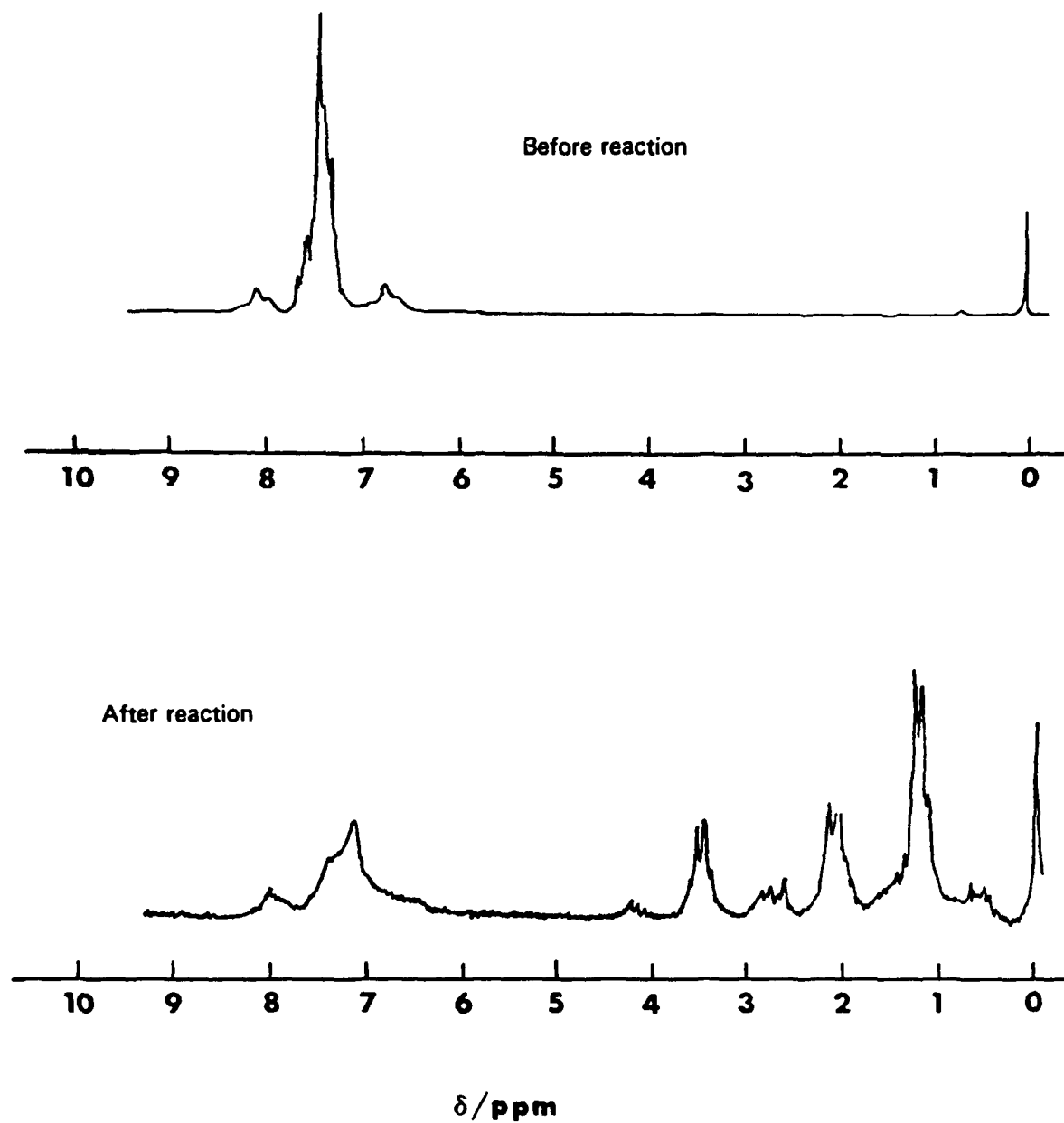


Figure 2. 90 MHz ¹H NMR spectra of Aroclor 6050 in CDCl₃ (relative to TMS).

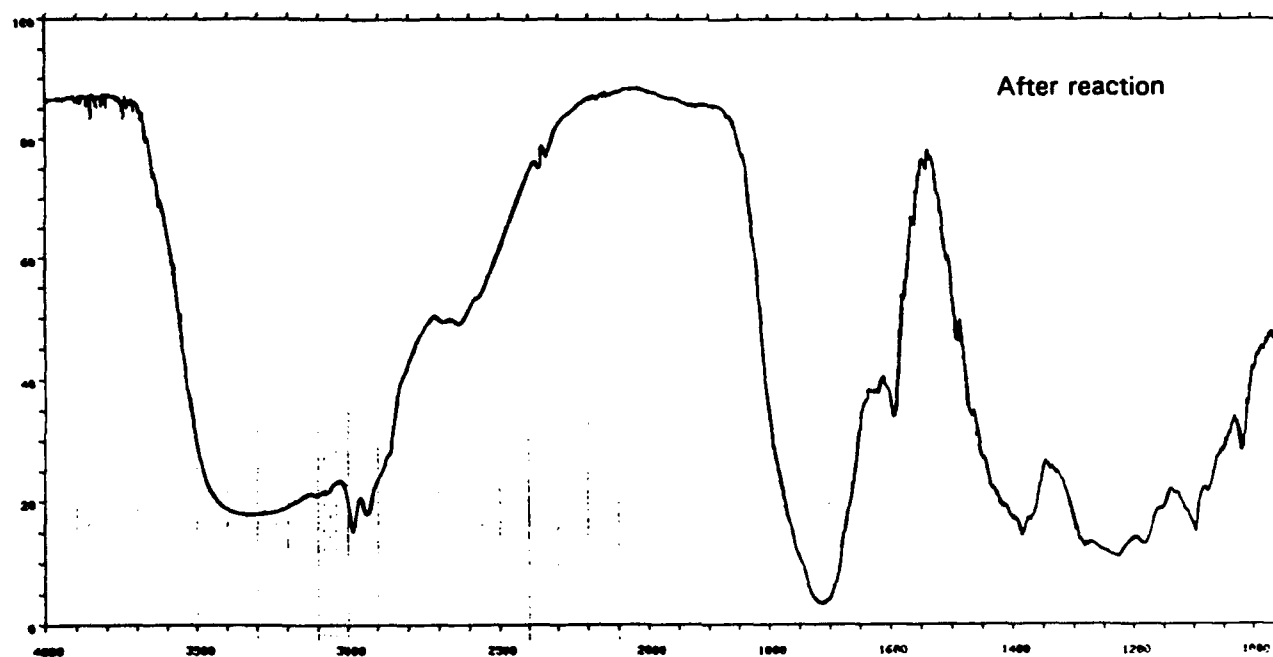
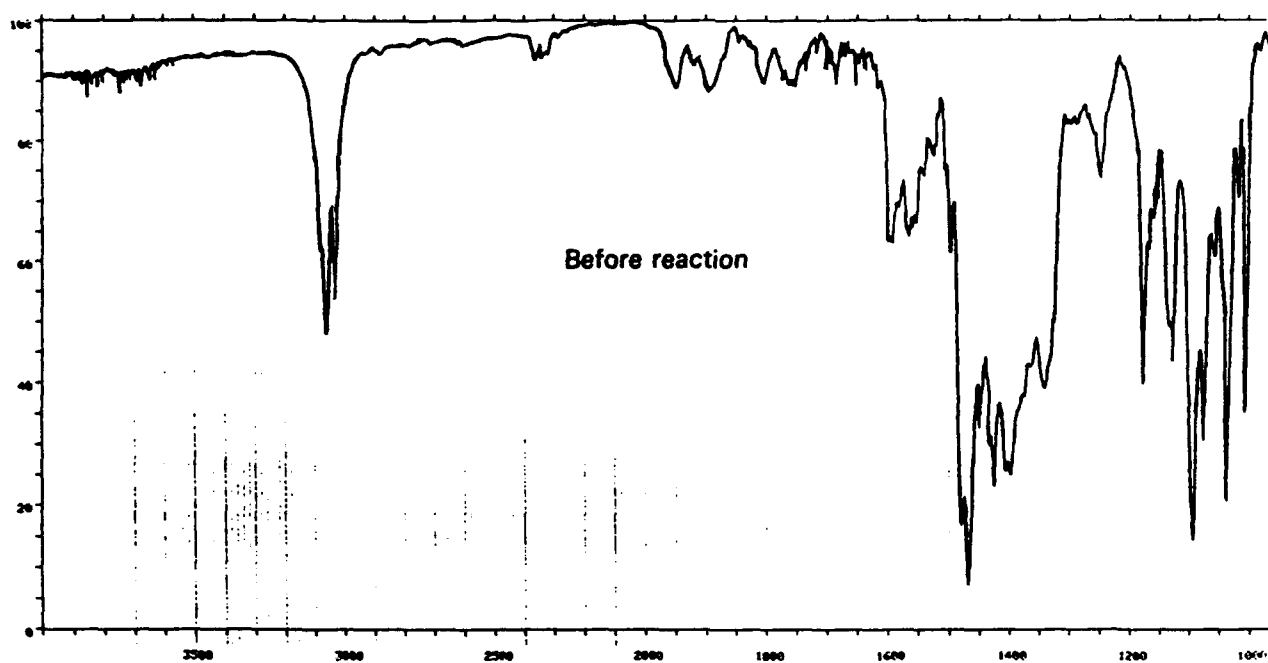


Figure 3. Infrared spectra of Arochlor 6050.

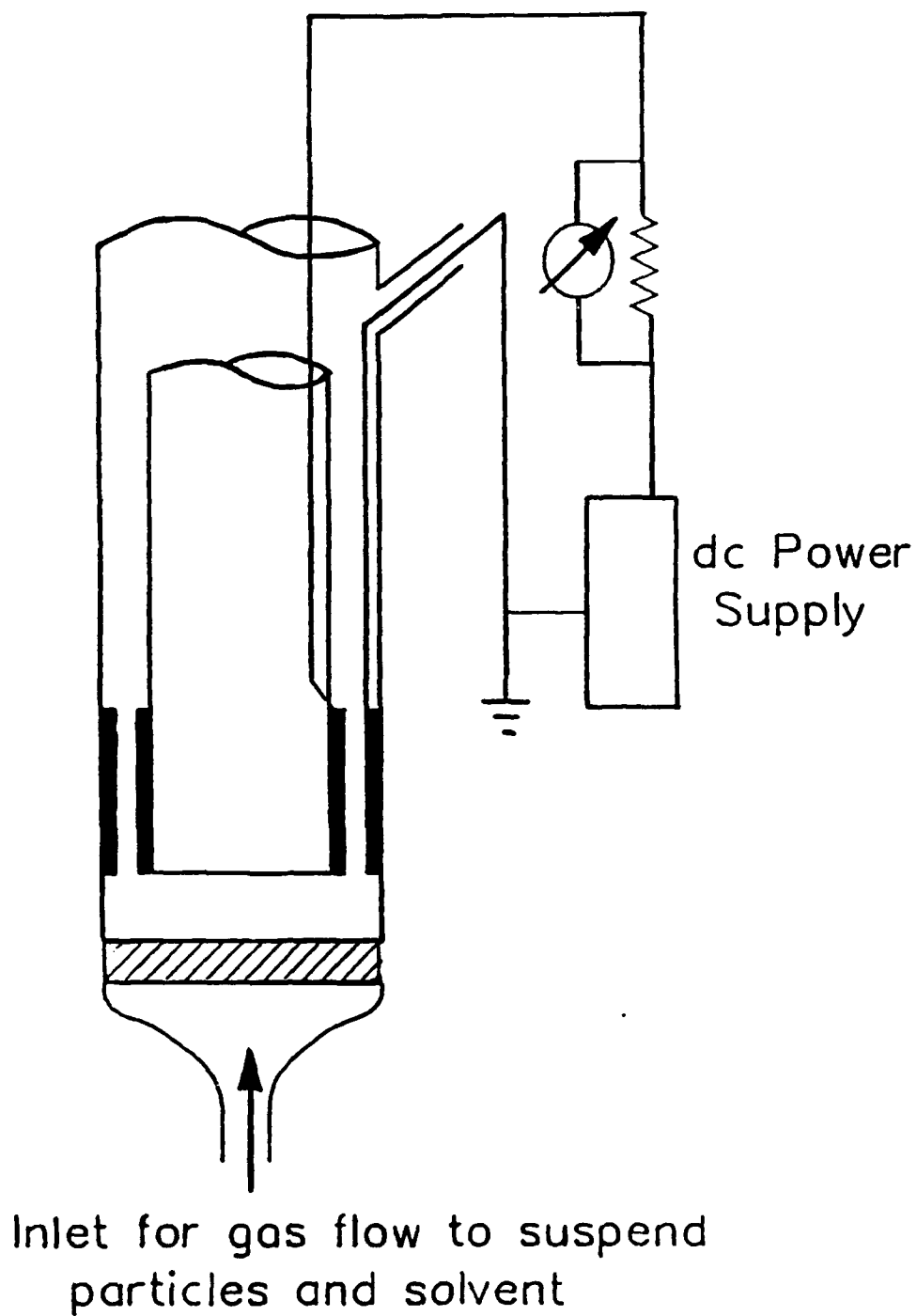


Figure 4. Schematic of the dispersion electrolysis cell and the electronic circuitry.

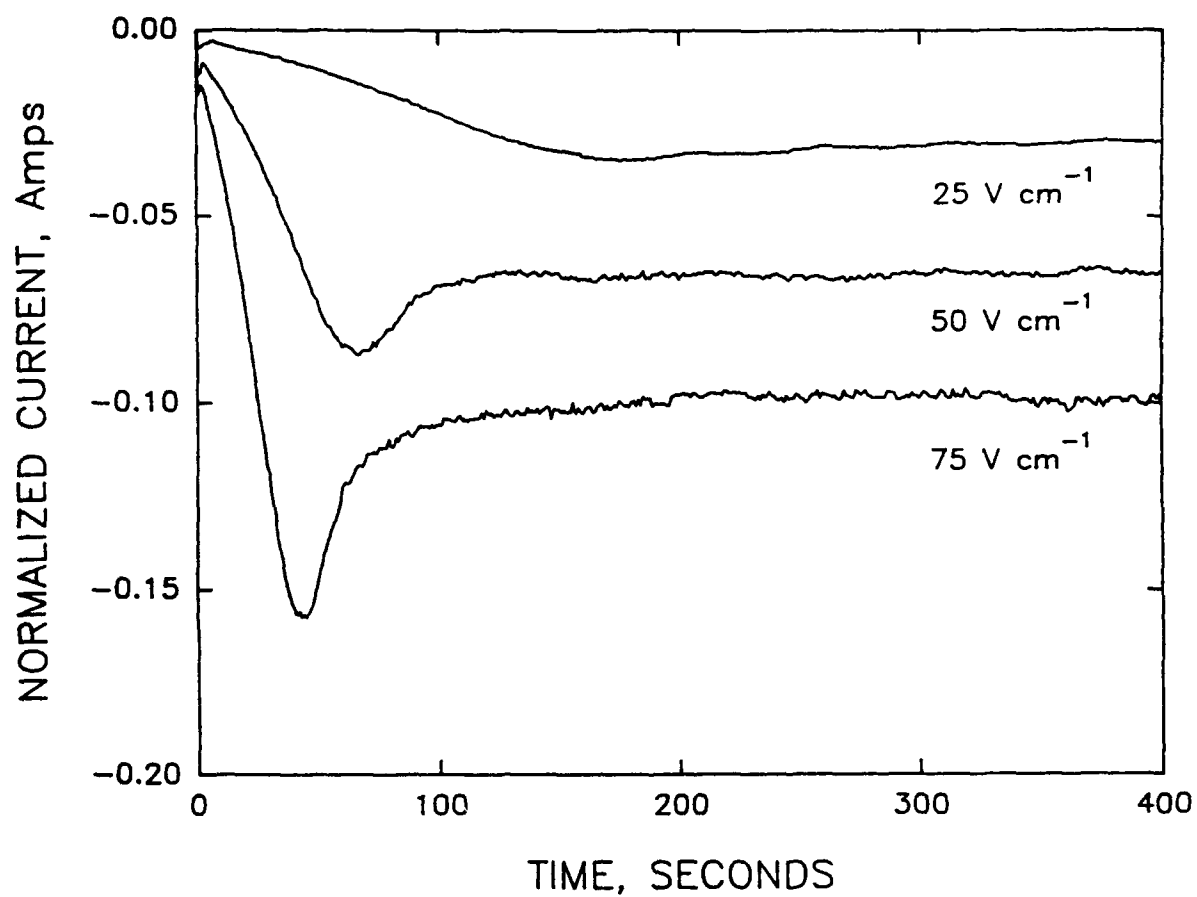


Figure 5. Current-time profile as a function of the applied voltage gradient for dispersions of NaY in 18 M Ω -cm water. The cell current has been normalized to the suspension density (~ 2.5 mg/cm³); cell gap = 0.4 cm; platinum feeder electrodes.